

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization International Bureau



(43) International Publication Date
29 July 2004 (29.07.2004)

PCT

(10) International Publication Number
WO 2004/062836 A2

(51) International Patent Classification⁷:

B22D

SI, SK, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

(21) International Application Number:

PCT/US2003/041577

Declarations under Rule 4.17:

(22) International Filing Date:

30 December 2003 (30.12.2003)

— as to applicant's entitlement to apply for and be granted a patent (Rule 4.17(ii)) for the following designations AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW, ARIPO patent (BW, GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG)

(25) Filing Language:

English

(26) Publication Language:

English

(30) Priority Data:

10/339,523 9 January 2003 (09.01.2003) US

— as to the applicant's entitlement to claim the priority of the earlier application (Rule 4.17(iii)) for the following designations AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW, ARIPO patent (BW, GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG)

(71) Applicant (for all designated States except US): HENKEL KOMMANDITGESELLSCHAFT AUF AKTIEN [DE/DE]; 67 Henkelstrasse, 40589 Duesseldorf (DE).

(72) Inventors; and

(75) Inventors/Applicants (for US only): BOTZ, Frank, K. [US/US]; 701 Mike Drive #20, Spartanburg, SC29303 (US). KUTZKO, Paul, B. [US/US]; 11901 Arcola, Livonia, MI 48150 (US).

(74) Agent: HARPER, Stephen, D.; Henkel Corporation, Patent Department, The Triad, Suite 200, 2200 Renaissance Boulevard, Gulph Mills, PA 19406 (US).

(81) Designated States (national): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW.

(84) Designated States (regional): ARIPO patent (BW, GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG)

Published:

— without international search report and to be republished upon receipt of that report

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: LUBRICANTS SUITABLE FOR HYDROFORMING AND OTHER METAL MANIPULATING APPLICATIONS

(57) Abstract: The present invention discloses a hydroforming process for metal parts that uses liquid-film and solid-film lubricants. The lubricants used in the invention are particularly useful for die-side lubrication. The process includes a step in which a ductile metal part is over-coated with either the liquid-film or solid-film lubricant. The liquid lubricants preferably include an oil and a optionally a surfactant. The solid lubricants preferably include a hard wax and optionally a surfactant.

WO 2004/062836 A2

LUBRICANTS SUITABLE FOR HYDROFORMING AND OTHER METAL MANIPULATING APPLICATIONS

CROSS-REFERENCE TO RELATED APPLICATION

This application is a continuation-in-part of U.S. Application Serial
5 No. 09/957,911 filed September 21, 2001 which, in turn, claims the benefit of U.S.
Provisional Application Serial No. 60/234,833, filed September 22, 2000.

BACKGROUND OF THE INVENTION

1. Field of the Invention

This invention relates to lubricants used in metal forming processes
10 and, in particular, to lubricants used in hydroforming processes.

2. Background Art

Processes in which metal parts are manipulated or formed typically
require lubricants to reduce equipment wear. These processes include such
operations as bending, swaging, roll-tapping, drawing, and hydroforming.
15 Hydroforming is a particularly important process in which a relatively complex
metal part is fabricated.

There are two types of hydroforming processes. One is used to form
parts from sheet metal and the other is used to form parts from metal tubes. Many
tube hydroforming applications are currently utilized by the automotive industry.

20 In a tube hydroforming process, a workpiece tube is placed in a tool
cavity. The geometry of the die cavity corresponds to the external geometry of the
produced part. The tool cavity is closed by the ram movement of a press. At the
same time, the tube ends are loaded by two punches moving along the tube axis, and

an aqueous fluid is pumped into the tube. As the internal pressure of this pressure-side aqueous fluid is increased, the tube expands until the expanding tube wall contacts the inner surface of the die, and the part is formed.

5 There are three types of lubricants involved in the tube hydroforming process: a bending lubricant, the pressure-side aqueous fluid mentioned above, and a die-side lubricant that is used between the workpiece tube and the die. The bending lubricant is used on the inside of the tube to bend the tube into a desired shape just prior to mounting the tube in the hydroforming tool cavity. The pressure-side fluid is the aqueous hydraulic fluid used to transmit the pressure to the inside
10 of the tube. Although little lubricity is required of the pressure-side fluid, other properties, such as corrosion protection, high pressure stability, and the ability to reject the bending and die-side lubricants, are important to the performance. The die-side lubricant is the primary forming fluid in high-pressure hydroforming. It provides the lubricity between the workpiece and the die.

15 The demands on the die-side lubricant vary widely. Some light duty applications require little of the die-side lubricant. In the case of lower pressure applications, the pressure-side fluid may also be used simultaneously to transmit pressure inside the tube and to provide die-side lubrication. As the complexity of the application increases, the importance of the die-side lubricant increases.
20 Furthermore, the die-side lubricants' compatibility with the pressure-side lubricant and the removal of the die-side lubricant from the newly formed part are important considerations.

SUMMARY OF THE INVENTION

25 The present invention discloses a liquid film die-side hydroforming lubricant that comprises an oil and a surfactant. The liquid film die-side lubricant is typically already a liquid when applied to the workpiece tube. Preferably, the liquid film die-side lubricant has lubrication properties that are not substantially

damaged by contact with the pressure-side fluid which usually contains water. Furthermore, the liquid film die-side lubricant preferably has high viscosity.

In accordance with another aspect of the present invention, a solid film die-side hydroforming lubricant is disclosed. The solid film die-side lubricant 5 comprises a wax such that the stress value within the die-side lubricant is at least 540 kPa at 0.75 sec after a compressive stress is imposed. Preferably, the solid film die-side lubricant is a liquid when applied to the workpiece tube. The applied liquid then either dries or cures into a solid lubricating film. The solid film die-side lubricant has lubrication properties that are not substantially damaged by contact 10 with the pressure-side fluid, which usually contains water. Furthermore, the liquid film die-side lubricant preferably has high viscosity. When the die-side lubricant of the present embodiment is a solid at the time of emplacement, the die-side lubricant preferably has high hardness and optionally a high elasticity. The solid film lubricant also optionally includes a wetting agent to improve the ability of the 15 composition to wet metallic surfaces.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

Reference will now be made in detail to presently preferred compositions or embodiments and methods of the invention, which constitute the best modes of practicing the invention presently known to the inventor.

20 For purposes of the present invention, the resistance of a lubricant to damage to its lubrication properties by pressure-side fluid is most conveniently measured by measuring the coefficient of friction of two metal surfaces, lubricated with the die-side lubricant to be measured, in a sliding friction test at a pressure from 65 to 400 bars and in a twist compression test at a pressure from 675 to 2500 25 bars. A die-side lubricant to be tested is first placed on one surface of a substrate of the same type of metal as is to be hydroformed in the same manner as if the substrate were to be hydroformed, but the substrate in this instance has a shape suitable for the intended method of measurement of coefficient of friction. After the

coefficient of friction has been measured, the die-side lubricant layer is sprinkled or otherwise gently wet with the intended pressure-side fluid for hydroforming or a surrogate for this pressure-side fluid, plain deionized or tap water often being an effective surrogate. A volume of the pressure-side fluid or surrogate therefor that

5 is not more than about twice the volume of the wetted die-side lubricant film itself should be used, and no substantial mechanical force such as would result from high pressure spraying should be used. After a minute or two of contact between the lubricant layer and the pressure-side fluid or surrogate therefor, any remaining aqueous liquid is allowed to drain away under the influence of natural gravity, and

10 the coefficient of friction of the substrate bearing the thus-drained die-side lubricant film is again measured. The die-side lubricant has sufficient pressure-side fluid-resistance for the purposes of this invention when the coefficient of friction measured with the thus wetted and drained die-side lubricant film does not exceed the coefficient of friction measured under the same conditions with the originally

15 emplaced and unwetted die-side lubricant film by an amount that is preferably more than about 50 percent of the value of the coefficient of friction for the originally emplaced and unwetted die-side lubricant film, more preferably more than about 30 percent of the value of the coefficient of friction for the originally emplaced and unwetted die-side lubricant film, and most preferably more than about 1.0 percent

20 of the value of the coefficient of friction for the originally emplaced and unwetted die-side lubricant film. In particularly favorable instances, the coefficient of friction is reduced by contact with the pressure-side fluid or surrogate therefor. All of the measurements involved in this determination of the pressure-side fluid resistance of a lubricant should be made at the intended temperature of the hydroforming process

25 itself, or, if the latter is unknown, at a normal ambient human comfort temperature (between 18 and 23° C).

In one embodiment of the present invention, a liquid film die-side hydroforming lubricant is disclosed. The liquid film die-side lubricant is typically already a liquid when applied to the workpiece tube. The liquid film die-side lubricant has lubrication properties that are not substantially damaged by contact with the pressure-side fluid as defined above. Furthermore, the liquid film die-side hydroforming lubricant includes an oil that has a kinematic viscosity measured at

40° C, that is at least, with increasing preference in the order given, 2.5, 5.0, 7.5, 10.0, 12.5, 15.0, 17.5, or 20 stokes. Suitable commercially available oils include vegetable oils, blown (alternatively called "oxidized") vegetable oils, polymers of vegetable oils, animal oils, and blown animal oils along with typical petroleum oils.

5 Specific examples include blown canola oil, blown fish oil, canola oil, blown rapeseed oil, and naphthenic oil.

The liquid film die-side hydroforming lubricant ("liquid film composition") of the present invention optionally further include a surfactant. The surfactant improves the cleaning properties of the lubricant, i.e., the ease of removing residual lubricant. Although any surfactant may be utilized, preferably non-ionic surfactants are used. The surfactant also preferably improves the lubricity of the liquid film when wetted. Though not restricting the improvement of lubricity to any particular mechanism, the surfactant appears to form an emulsified layer when wetted that enhances lubricity. However, the amount of surfactant is not so much that the liquid film is deteriorated during emulsification. The surfactant is preferably present in an amount of 0.1% to 10% of the total weight of the liquid film composition, more preferably in an amount of 1.0% to 5% of the total weight of the liquid film composition, and most preferably in an amount of about 2.5% of the total weight of the liquid film composition. Preferred surfactants include vegetable oil ethoxylates, ethoxylates of alkyl alcohols, ethoxylates of acetylenic diols, block copolymers of ethylene and propylene oxides, ethoxylates of alkyl carboxylates such as typical fatty acids, alkyl polyglycosides, and mixtures thereof. Examples include but are not limited to Chemal DA-6, Chemal DA-9, Chemal LA-4, Chemax CO-5, Chemax CO-16, Chemax CO-25, Chemax CO-30, Chemax CO-20, Chemax CO-36, Chemax CO-40, Chemax CO-80, and Chemax CO-200/50 commercially available from Chemax, Inc. located in Greenville, SC. Suitable surfactants also include but are not limited to Surfynol 440 commercially available from Air Products, TOMAH E-14-5 (poly (5) oxyethylene isodecyloxypropylamine) and TOMAH E-14-2 commercially available from Tomah Products Inc. located in Milton, WI; NINOL 11CM (a modified coconut diethanolamide surfactant sold by Stepan, Inc.) TRITON X-100 (octylphenol ethylene oxide condensate; Octoxynol-9) commercially available from Union Carbide; and APG 325 CS (decyl polyglucoside)

commercially available from Cognis Corporation located in Cincinnati, Ohio. Other suitable non-ionic surfactants include block surfactants containing polyoxypropylene hydrophobe(s) and polyoxyethylene hydrophile(s). In order to properly function, the surfactant must be soluble or dispersible in the lubricant. The blocks may be 5 homopolymeric or copolymeric, for example copolymers derived from oxyalkylating with mixtures of ethylene oxide and propylene oxide. Such surfactants are available from numerous sources, including the Pluronic®, Tetronic®, and Pluronic® R polyether surfactants from BASF Corporation.

10 In another embodiment of the present invention, a solid film die-side hydroforming lubricant ("solid film composition") is disclosed. Typically, the solid film lubricant will be applied to a surface as a liquid which is subsequently dried and cured. The resultant solid lubricant of the present invention preferably has a hardness as measured at 23-26° C by the American Society for Testing and Materials ("ASTM") Procedure Number D-5 that is not more than, with increasing 15 preference in the order given, 50, 40, 30, 20, 15, 13, 11, 9, 7, 5, or 3. The solid film lubricant of the present invention includes solid lubricants that are characterized by one or more of the following properties when subjected to a compressive stress within the range from 1.50 to 2.00 percent over a time interval of 0.20 to 0.30 seconds at 23-26° C:

20 - the stress value within the solid die-side lubricant 0.75 sec after the compressive stress began to be imposed is at least, with increasing preference in the order given, 500, 510, 520, 530, 540, 550, 560, 570, or 580 kiloPascals (this unit of stress being hereinafter usually abbreviated as "kPa");

25 - the stress value within the solid die-side lubricant 100 sec. after the compressive stress began to be imposed is at least, with increasing preference in the order given, 300, 350, 400, 450, 500, 510, 520, 530, 540, or 550 kPa; and

30 - the residual stress within the solid die-side lubricant 100 sec after the compressive stress began to be

imposed is at least, with increasing preference in the order given, 75, 80, 82, 84, 86, 88, or 90 percent of the maximum stress induced within the solid lubricant at any time up to 100 sec after the stress began to be imposed.

5

A method of measuring these stress values is described by T H. Sheilhammer, T.R. Rumsey, and J. M. Krochia in "Viscoelastic Properties of Edible Lipids," JOURNAL OF FOOD ENGINEERING 33 (1997), pages 305 - 320. This paper is hereby incorporated herein by reference to the extent that it is not inconsistent with any explicit statement herein. Preferred solid film lubricants include carnauba wax; candelilia wax; montan wax; microcrystalline waxes; solid alcohols, particularly primary alcohols having at least 18 carbon atoms per molecule; solid esters, particularly esters of primary alcohols having at least 18 carbon atoms per molecule with organic acids, especially unbranched monoacids, having at least 18 carbon atoms per molecule; and oxidized petroleum waxes.

10

15

The solid film die-side hydroforming lubricant of the present invention optionally further includes a surfactant. Although any surfactant may be utilized, preferably non-ionic surfactants are used. The surfactant also preferably improves the lubricity of the solid film when wetted. The surfactant is preferably present in an amount of 0.05% to 10% of the total weight of the solid film composition, more preferably in an amount of 0.1% to 5% of the total weight of the solid film composition, and most preferably in an amount of about 1% of the total weight of the solid film composition. Preferred surfactants include vegetable oil ethoxylates, ethoxylates of alkyl alcohols, ethoxylates of acetylenic diols, block copolymers of ethylene and propylene oxides, ethoxylates of alkyl carboxylates such as typical fatty acids, alkyl polyglycosides, and mixtures thereof. Suitable surfactants also include but are not limited to Surfynol 440 commercially available from Air Products, TOMAH E-14-5 (poly (5) oxyethylene isodecyloxypropylamine) and TOMAH E-14-2 commercially available from Tomah Products Inc. located in Milton, WI; NINOL 11CM (a modified coconut diethanolamide surfactant sold by Stepan, Inc.) TRITON X-100 (octylphenol ethylene oxide condensate; Octoxynol-9) commercially available from Union

Carbide; and APG 325 CS (decy1 polyglucoside) commercially available from Cognis Corporation located in Cincinnati, Ohio. Other suitable non-ionic surfactants include block surfactants containing polyoxypropylene hydrophobe(s) and polyoxyethylene hydrophile(s). The blocks may be homopolymeric or 5 copolymeric, for example copolymers derived from oxyalkylating with mixtures of ethylene oxide and propylene oxide. Such surfactants are available from numerous sources, including the Pluronic®, Tetronic®, and Pluronic® R polyether surfactants from BASF Corporation.

The solid film die-side lubricant optionally comprises a wetting agent. 10 Utilization of such agents improves the ability of the dry film composition (which is a liquid when applied) to wet metals such as the various steel alloys (stainless steel, hot rolled steel, and cold rolled steel), aluminum alloys, titanium, and copper. It will be recognized by those skilled in the art, that many wetting agents are surfactants and many surfactants are wetting agents. Accordingly, a subset of 15 the surfactants listed above will also function as wetting agents. Suitable wetting agents include, but are not limited to, nonionic fluorosurfactants, anionic fluorosurfactants, ethoxylated tetramethyldecynediols, acetylenic glycol-based surfactants, dialkylsulfosuccinates, and mixtures thereof. Suitable ethoxylated tetramethyldecynediols include members of the Surfynol 400 series such as Surfynol 20 440 and 420 commercially available from Air Products. An exemplary acetylenic glycol-based surfactant is Dynol 604 commercially available from Air Products. Suitable dialkylsulfosuccinates include dioctylsulfosuccinates. The preferred wetting agent is a fluorosurfactant which includes both nonionic fluorosurfactants and an anionic fluorosurfactants. Most preferably the wetting agent is a nonionic 25 fluorosurfactant. Suitable nonionic fluorosurfactants include fluoroaliphatic ethoxylates and related derivatives. Specifically, Clariant Fluowet OTN and DuPont Zonyl FSN 100 are nonionic surfactants that performed well. Fluowet OTN is a proprietary fluoroaliphatic ethoxylate commercially available from Clariant. Zonyl FSN 100 is a Telomer B monoether with polyethylene glycol which is a 1:1 mixture 30 of poly(oxy-1,2-ethandiyl), α -hydro- Ω -hydroxy-ether with α -fluoro- Ω -(2-

hydroxyethyl)poly(difluoromethylene). Suitable anionic fluorosurfactants include fluoroalkylsulfonates and carboxylates with a range of counter ions that include potassium, sodium, and amines. Preferably, the fluorosurfactant is present in an amount of about 0.1% to 1.0% by weight of the dry film composition. More 5 preferably, the fluorosurfactant is present in an amount of about 0.1% to 0.5% by weight of the dry film composition.

The solid film die-side lubricant also optionally includes a corrosion inhibitor and/or a defoamer. Suitable defoamers include neo-decanoic acid. Suitable 10 corrosion inhibitors include soaps or salts of carboxylic acids or organo-sulfonates. Agents capable of adjusting the pH of the lubricant may also be included, such as, for example, amines (e.g., alkanolamines).

Regardless of whether the die-side lubricant is solid or liquid at the time of emplacement or whether the die-side lubricant has an aqueous-based liquid after being emplaced, the coefficient of sliding friction between two metal surfaces 15 with a layer between them of a die-side lubricant to be used in a process according to the invention preferably is not more than about 0.3 to 0.5, more preferably is not more than about 0.1 to 0.3, and most preferably is not more than about 0.04 to 0.1.

Furthermore, the die-side lubricant is preferably capable of being 20 readily cleaned from the hydroformed object after hydroforming is complete, preferably with an aqueous-based cleaner. Preferably, the die-side lubricant is capable of being cleaned at a temperature not higher than 55 °C, more preferably a temperature not higher than 40 °C, and most preferably at a temperature not higher than 28 °C. This preference is not inconsistent with the need for pressure-side fluid resistance of the die-side lubricant as described above. Typical aqueous based 25 cleaners are either more acidic or more alkaline than most aqueous pressure-side fluids used in hydroforming. Furthermore, even if the cleaners are neutral, they usually contain other cleaning-promoting ingredients such as detergents surfactants

that are not present in typical pressure-side fluids for hydroforming.

The die-side lubricant is also preferably easy to separate from the pressure-side fluid should the lubricant become contaminated by the pressure-side fluid. Accordingly, self-segregation of the die-side lubricant into a separate phase 5 that can be skimmed or drained off from a reservoir of pressure-side fluid is highly desirable.

Finally, the lubricant is preferably easy to apply to the surface to be lubricated, without producing any hazard such as flammable, toxic, or noxious fumes, without requiring any equipment more complicated than simple spray, 10 immersion, and/or roll coating, and without requiring any special drying equipment. For example, if a die-side lubricant that is a solid when emplaced ready for use can be applied from a latex and allowed to dry in the ambient air without producing any fire hazard or unpleasant odor, there is a substantial practical advantage and therefore a preference for it over a solid die-side lubricant that must be melted to be 15 applied and then quickly cooled to avoid having the melted die-side lubricant run off the substrate being hydroformed.

In another embodiment of the present invention, a process for hydroforming a tube of a ductile solid material is provided. The process comprises the following steps:

20 (I) providing a pressure-side fluid and an openable die having an interior surface of a shape to which it is desired to have the hydroformed part of the outer surface of the tube of ductile solid material conform after the tube has been hydroformed;

25 (II) forming over the outer surface of the tube of ductile solid material a coating of a die-side lubricant suitable for use in a process according to the invention as described above, so as to form a coated ductile tube;

- (III) emplacing the coated ductile tube within at least a part of said openable die and closing the die, so that a portion of the outer surface of the ductile tube that is desired to be hydroformed is within the closed openable die;
- 5 (IV) filling the interior of the tube of ductile solid with a volume of said pressure-side fluid, so that said pressure-side fluid exerts equal pressure on all parts of the internal surface of the tube of ductile solid with which the pressure-side fluid is in physical contact; and
- 10 (V) applying to said volume of pressure-side fluid filling said interior of the ductile tube, while the ductile tube remains emplaced within the closed openable die as recited in operation (III) above, a sufficient pressure to cause at least a portion of the outer surface of the coated ductile tube to conform to the inner surface of the closed openable die.
- 15

Only a relatively thin layer of the die-side lubricant is needed for satisfactory lubrication. More particularly, the average thickness of the die-side lubricant layer formed before hydroforming begins preferably is in the range 0.2 to 200 microns, more preferable in the range 1.0 to 100 microns, and most preferably 20 about 15 microns. Uniformity of the die-side lubricant is not critical. The films may even be discontinuous ball-like lumps and aggregates evenly distributed over the surface of the part.

Preferred lubricants for use according to the invention can be readily removed from surfaces of metal ductile tubes, after hydroforming is completed, by 25 conventional alkaline cleaners.

Except for use of the characteristic lubricant for this invention as described above, the process conditions for a hydroforming process according to the

invention are normally the same as those already in use in the art. A process according to the invention is particularly advantageous in "high pressure" hydroforming, in which the hydraulic pressure in step (V) of the process as described above is at least 340 bars and independently is particularly advantageous 5 in hydroforming cold rolled steel, but is suitable for hydroforming any other ductile solid as well. Hydroforming with these lubes is successful with hot-rolled steel, cold-rolled steel, and aluminum, both 5000 and 6000 series alloys.

The invention may be further appreciated by consideration of the following examples and comparison examples. In all of the tests below, the metal 10 substrate was type ADKQ 95 hot-rolled steel, which is one of the most commonly hydroformed substrates.

TEST METHODS

CORNERFILL TEST

The cornerfill test is designed to test the properties required by a die- 15 side hydroforming lubricant in the expansion zone of a hydroforming process. In these tests, the exterior surface of a welded cylindrical steel tube was coated with test die-side lubricant and then mounted in a die with a square cross-section that was within one millimeter of touching the exterior cross-section of the cylindrical steel tube at the center of all four walls of the square die, with no weld line at or near one 20 of these centers of the die walls. The lubricant-coated exterior of the steel tube was then sprayed lightly with water before the die was closed. The interior of the steel tube was then filled with a volume of a water-based pressure side fluid, and the pressure in the tube was then increased until the tube burst. Sensors detected the pressure at various stages of expansion, the maximum pressure before the tube 25 burst, and the maximum expansion of the tube. The burst tube was then removed from the die, and the tube burst location was noted. Then the dimensions of the burst tube were measured and the true thickness strain was calculated for seven

locations: the four corners and the centers of the three walls of the square cross-section into which the tube had expanded that did not include the original welded area. Three of the properties measured in this type of test are generally considered relevant to performance in actual hydroforming. A higher burst pressure is better
5 than a lower one; a low standard deviation of the true thickness strain is better than a higher one; and a burst near the center of the tube is better than a burst in any other part of the tube.

TWIST COMPRESSION TEST

A twist compression test is designated to test the properties required
10 by a die-side hydroforming lubricant in transition zones near the edges of expansion zones in hydroforming. In these tests, an annular tool was rotated under pressure over a flat plate of steel on which the test lubricant had been emplaced. The pressure applied on the lubricated plate in one set of tests was 10,000 psi and in another was 15,000 psi. These pressures are typical of commercial hydroforming
15 of hot rolled steel tubes. A plot of the coefficient of friction as a function of time was generated. The results are reported at 1, 2, and 3 revolutions. The test was first conducted dry for each lubricant and then twice after the lubricant had been sprayed lightly with water. Only the average of the latter two of these measurements is reported below. The tests were also performed on lubricants
20 sprayed with Novacool 9034, a pressure-side fluid commercially available from Henkel Corporation, Madison Heights, Michigan. The lower the coefficient of friction in these tests, the better performance the lubricant usually gives in the transition zone.

SLIDING FRICTION TEST

25 The sliding friction test measures the properties of the die-side lubricant that are important in the "end-feeding zone" of a hydroforming process. In this end-feeding zone, the tube being hydroformed does not substantially expand

or contract its eternal cross-section, although its walls may thin or thicken. Instead, part of the tube moves laterally along the die to allow for expansion in another part of the die. This end-feeding is very important in the production of some part designs by hydroforming. The procedure used for this type of test for which values 5 are reported here is described in American Society for Testing and Materials (“ASTM”) Procedure 4173-82, using a compressive pressure between the sliding workpieces of 69 bars (1000 psi). (This is officially an “obsolete” ASTM test method, but it is still useful for measuring the coefficient of friction in sliding friction.) The lower the coefficient of friction in sliding friction, the better is the 10 lubricant in the end-feeding zone.

Solid Film Lubricants

Examples 1-4 provides examples of the solid film lubricants of the present invention.

Example 1

15

Component	Weight %
carnauba wax aqueous emulsion, 22% solids, (Michelman Michem Lube 160)	89.5%
water	8.0%
monoethanolamine	0.5%
sodium benzoate	2.0%.
Total	100%

20

25

The monoethanolamine reduces the staining by the wax by the slightly acidic carnauba wax by raising the pH. Finally, the sodium benzoate is a

corrosion inhibitor. The carnauba wax is characterized with a hardness of about 1 (ASTM-D-5), a particle size of about 0.15 microns, and a melting point of about 85 °C.

5

Example 2

10

Component	Weight %
microcrystalline wax emulsion, 42% solids, (Michelman Michem Lube 124)	99.0
nonionic surfactant, (Air Products Surfynol 440)	1.0
Total	100%

15

The microcrystalline wax is a mixture of two waxes of hardness 5 and 13 using ASTM D-5 and with melting points centered around 68 and 101 degrees C. Furthermore, the microcrystalline wax has a particle size of about 0.18 microns.

20

Example 3

25

Component	Weight %
Fischer-Tropsch wax emulsion, 40% solids, (Michelman Emulsion 64540)	99.9
nonionic surfactant, (Air Products Surfynol 420)	0.1

Total	100%
-------	------

1 The Fischer-Tropsch wax is characterized with a hardness of about
 1 (ASTM-D-5), a particle size of about 0.6 microns, and a melting point of about
 98 °C.

5

Example 4

10

15

Component	Weight %
Fischer-Tropsch wax emulsion, 40% solids, (Michelman Emulsion 98040M1)	92.5
nonionic surfactant, (Air Products Surfynol 440)	1.0
neodecanoic acid	4.0
KOH, 45%	2.5
Total	100.00

20 The neodecanoic acid functions as both a corrosion inhibitor and defoamer. The Fischer-Tropsch wax is characterized as set forth above for example 3.

20

The results of the twist compression measurements for the lubricants in examples 1-3 are summarized in Tables 1 and 2.

Table 1. Twist Compression Results using 6061 T4 Aluminum at 10,000 psi

Lube	Prewet Fluid	Initial COF	COF @ 1 rev	COF @ 2 rev	COF @ 3 rev
------	--------------	-------------	-------------	-------------	-------------

example 1	water	0.07	0.06	0.07	0.09
example 1	9034	0.01	0.03	0.03	0.04
example 2	water	0.01	0.03	0.06	0.08
example 2	9034	0.01	0.03	0.07	0.10

5

Table 2. Twist Compression Results using Hot-Rolled Steel at 15,000 psi

10

Lube	Prewet Fluid	initial COF	COF @ 1 rev	COF @ 2 rev	COF @ 3 rev
example 1	water	0.01	0.02	0.04	0.06
example 1	9034	0.04	0.04	0.05	0.05
example 2	water	0.01	0.03	0.03	0.04
example 2	9034	0.01	0.03	0.03	0.03
example3	water	0.01	0.02	0.03	0.04
example3	9034	0.01	0.02	0.03	0.04

15

The coefficient of friction (COF) was determined by the sliding friction test for various wetted and unwetted waxes. The results are summarized in Table 3. Surprisingly, the COF is reduced when the waxes are wetted.

Table 3. Coefficient of friction for various waxes.

20

Wax	COF (Neat)	COF (wetted)
carnauba wax	0.30-0.20	0.20-0.10
montan wax	0.20-0.18	0.18-0.12
microcrystalline wax	0.12-0.10	<0.10
Fischer-Tropsch wax	-	<0.10

Liquid Film Lubricants

Examples 5-8 provide examples of the liquid film compositions of the present invention.

Example 5

5

Component	Weight %
blown canola oil, Z2	97.5
viscosity	
ethoxylated castor oil, Chemax CO-5	2.5
Total	100.00

10

In view of the tackiness of blown canola oil, the ethoxylated castor oil is water miscible and makes it easier to wash the composition in example 5. The ethoxylated castor oil is provided in such an amount that the washability of the formulation improved but lubricity of the formulation is only minimally degraded. 15 Small amounts of the ethoxylated castor oil actually improve lubricity. Example 5 has a burst pressure of about 10,510 psi; a twist compression coefficient of friction at 10,000 psi of about 0.06; a twist compression coefficient of friction at 15,000 psi of about 0.05; and a sliding coefficient of about 0.065.

20

Tables 4 and 5 summarize the twist compression results for the composition described by example 5.

Table 4. Twist Compression Results using 6061 T4 Aluminum at 10,000 psi

Lube	Prewet Fluid	initial COF	COF @ 1 rev	COF @ 2 rev	COF @ 3 rev
example 5	water	0.06	0.27	0.29	0.28

example 5	9034	0.10	0.24	0.32	0.35
-----------	------	------	------	------	------

Table 5. Twist Compression Results using Hot-Rolled Steel at 15,000 psi

5

Lube	Prewet Fluid	initial COF	COF @ 1 rev	COF @ 2 rev	COF @ 3 rev
example 5	water	0.08	0.07	0.09	0.14
example 5	9034	0.09	0.07	0.08	0.07

Example 6

10

Component	Weight %
canola oil	97.5
ethoxylated castor oil, Chemax CO-5	2.5
Total	100.00

15

Component	Weight %
blown herring oil, Z5 viscosity	95.0

ethoxylated castor oil, Chemax CO-5	5.0
Total	100.00

5

Example 8

10

Component	Weight %
blown canola oil, Z2 viscosity	47.5
naphthenic oil, 100 SUS viscosity	50.0
ethoxylated castor oil, Chemax CO-5	2.5
Total	100.00

15 Example 8 has a burst pressure of about 10,510 psi; a twist compression coefficient of friction at 10,000 psi of about 0.06; a twist compression coefficient of friction at 15,000 psi of about 0.05; and a sliding coefficient of about 0.065

20 The coefficient of friction (COF) was determined for mixtures of blown canola oil and various surfactant. The COF was measure both for neat (unwetted) and wetted mixtures. Table 6 summarizes the results. The coefficient of friction is surprisingly reduced in each case when wetted. Chemal DA-6 is the surfactant ethoxylated decyl alcohol with 6 moles of ethoxylation for each mole of alcohol, Chemal DA-9 is the surfactant ethoxylated decyl alcohol with 9 moles of ethoxylation for each mole of alcohol, Chemal LA-4 is the surfactant ethoxylated

lauryl alcohol with 4 moles of ethoxylation for each mole of alcohol, Chemax CO-5 is the surfactant ethoxylated castor glyceride with 5 moles of ethoxylation for each mole of castor glyceride; Chemax CO-16 is the surfactant ethoxylated castor glyceride with 16 moles of ethoxylation for each mole of castor glyceride; and Chemax CO-80 is the surfactant ethoxylated castor glyceride with 80 moles of ethoxylation for each mole of castor glyceride.

Table 6. COF for neat and wetted mixtures of blown canola oil and surfactant.

	Lubricant	COF at 2350 psi	% reduction in COF
10	blown canola oil + 2.5% DA-6	0.040	
15	blown canola oil + 2.5% DA-6	0.025	37.5
20	blown canola oil + 2.5% DA-9	0.032	
25	blown canola oil + 2.5% DA-9	0.028	12.5
30	blown canola oil + 2.5% LA-4	0.024	
	blown canola oil + 2.5% LA-4	0.017	29
	blown canola oil + 2.5% CO-5	0.037	
	blown canola oil + 2.5%CO-5	0.021	43
	blown canola oil + 2.5%CO-16	0.035	
	blown canola oil + 2.5%CO-16	0.022	37
	blown canola oil + 2.5%CO-80	0.036	
	blown canola oil + 2.5%CO-80	0.024	33

The COF was determined for neat (unwetted) and wetted mixtures of blown canola oil and the surfactant Chemax CO-40. Table 7 summarizes the COF for varying amounts of Chemax CO-40 in blown canola oil, Z2, viscosity. Chemax is an ethoxylated caster glyceride with 40 moles of ethoxylation for each mole of 5 caster glyceride. Again, the wetted mixtures have lower COF than the neat mixture.

Table 7. COF for neat and wetted mixtures of blown canola oil and Chemax CO-40.

	Lubricant	COF at 2900 psi	% reduction in COF	COF at 2900 psi	% reduction in COF
10	blown canola oil + 2.5%CO-40 (neat)	0.015	-	-	-
	blown canola oil + 2.5%CO-40 (wetted)	0.012	20%	-	-
15	blown canola oil + 2.5%CO-40 (neat)	-	-	0.034	-
	blown canola oil + 2.5%CO-40 (wetted)	-	-	0.023	32%
20	blown canola oil + 5%CO-40 (neat)	-	-	0.042	-
	blown canola oil + 5%CO-40 (wetted)	-	-	0.039	7%

While embodiments of the invention have been illustrated and described, it is not intended that these embodiments illustrate and describe all possible forms of the invention. Rather, the words used in the specification are 25 words of description rather than limitation, and it is understood that various changes may be made without departing from the spirit and scope of the invention.

WHAT IS CLAIMED IS:

- 1 1. A process for hydroforming a tube of ductile solid material,
2 the process comprising:
 - 3 (I) providing a pressure-side fluid and an openable die having an interior
4 surface of a shape to which it is desired to have the hydroformed part
5 of the outer surface of the tube of ductile solid material conform after
6 the tube has been hydroformed;
 - 7 (II) forming over the outer surface of the tube of ductile solid material a
8 coating of a die-side lubricant selected from the group consisting of:
 - 9 1. a liquid lubricant comprising an oil and a surfactant;
 - 10 2. a solid lubricant comprising a wax wherein the stress
11 value within the solid die-side lubricant 0.75 sec after
12 the compressive stress began to be imposed is at least
13 540 kPa; the stress value within the solid die-side
14 lubricant 100 sec. after the compressive stress began
15 to be imposed is at least 300 kPa; and the residual
16 stress within the solid die-side lubricant 100 sec after
17 the compressive stress began to be imposed is at least
18 75 percent of the maximum stress induced within the
19 solid lubricant at any time up to 100 sec after the
20 stress began to be imposed; and
 - 21 3. mixtures thereof.
 - 22 (III) emplacing the coated ductile tube within at least a part of said
23 openable die and closing the die, so that a portion of the outer
24 surface of the ductile tube that is desired to be hydroformed
25 is within the closed openable die;
 - 26 (IV) filling the interior of the tube of ductile solid with a volume
27 of said pressure-side fluid, so that said pressure-side fluid
28 exerts essentially equal pressure on all parts of the internal

29 surface of the tube of ductile solid with which the pressure-
30 side fluid is in physical contact; and
31 (V) applying to said volume of pressure-side fluid filling said
32 interior of the ductile tube, while the ductile tube remains
33 emplaced within the closed openable die as recited in
34 operation (III) above, a sufficient pressure to cause at least a
35 portion of the outer surface of the coated ductile tube to
36 conform to the inner surface of the closed openable die.

1 2. The process of claim 1, wherein the stress value within the solid
2 die-side lubricant 0.75 sec after the compressive stress began to be imposed is at
3 least 540 kPa.

1 4. The process of claim 1, wherein the stress value within the solid
2 die-side lubricant 100 sec. after the compressive stress began to be imposed is at
3 least 450 kPa.

1 5. The process of claim 1, wherein the stress value within the solid
2 die-side lubricant 100 sec. after the compressive stress began to be imposed is at
3 least 550 kPa.

1 6. The process of claim 1, wherein the residual stress within the solid
2 die-side lubricant 100 sec after the compressive stress began to be imposed is at least
3 82 percent of the maximum stress induced within the solid lubricant at any time up
4 to 100 sec after the stress began to be imposed.

1 7. The process of claim 1, wherein the residual stress within the solid
2 die-side lubricant 100 sec after the compressive stress began to be imposed is at least
3 90 percent of the maximum stress induced within the solid lubricant at any time up
4 to 100 sec after the stress began to be imposed.

1 8. The process of claim 1, wherein the oil is selected from the group
2 consisting of vegetable oils, blown vegetable oils, polymers of vegetable oils, animal
3 oils, and blown animal oils, and mixtures thereof.

1 9. The process of claim 1, wherein the oil is selected from the group
2 consisting of blown canola oil, blown fish oil, canola oil, blown rapeseed oil,
3 naphthenic oil, and mixtures thereof.

1 10. The process of claim 1, wherein the surfactant is a non-ionic
2 surfactant.

1 11. The process of claim 10, wherein the surfactant is selected from
2 the group consisting of vegetable oil ethoxylates, ethoxylates of alkyl alcohols,
3 ethoxylates of acetylenic diols, block copolymers of ethylene and propylene oxides,
4 ethoxylates of alkyl carboxylates, alkyl polyglycosides, and mixtures thereof.

1 12. The process of claim 10, wherein the surfactant is present in an
2 amount of about 0.1% to 10% of the total weight of the liquid film composition.

1 13. The process of claim 10, wherein the surfactant is present in
2 an amount of about 1.0% to 5% of the total weight of the liquid film composition.

1 14. The process of claim 10, wherein the surfactant is present in
2 an amount of about 2.5% of the total weight of the liquid film composition.

1 15. The process of claim 1, wherein the wax is selected from the
2 group consisting of carnauba wax, candelilla wax, montan wax, microcrystalline
3 waxes, solid alcohols, solid esters, and oxidized petroleum waxes.

1 16. The process of claim 1, wherein the wax is a primary alcohol
2 having at least 18 carbon atoms per molecule.

1 17. The process of claim 1, wherein the wax is an ester of a
2 primary alcohol having at least 18 carbon atoms per molecule with an organic acid.

1 18. The process of claim 1, wherein the organic acid is an
2 unbranched monoacid, having at least 18 carbon atoms per molecule.

1 19. The process of claim 1, wherein the solid lubricant further
2 comprises a surfactant.

1 20. The process of claim 19, wherein the surfactant is a non-ionic
2 surfactant.

1 21. The process of claim 19, wherein the surfactant is selected
2 from the group consisting of vegetable oil ethoxylates, ethoxylates of alkyl alcohols,
3 ethoxylates of acetylenic diols, block copolymers of ethylene and propylene oxides,
4 ethoxylates of alkyl carboxylates, alkyl polyglycosides, and mixtures thereof

1 22. The process of claim 19, wherein the surfactant is present in
2 an amount of about 0.05% to 10% of the total weight of the dry film composition.

1 23. The process of claim 19, wherein the surfactant is present in
2 an amount of about 1.0% to 5% of the total weight of the dry film composition.

1 24. The process of claim 19, wherein the surfactant is present in
2 an amount of about 1% of the total weight of the dry film composition.

1 25. The process of claim 1, wherein the solid lubricant further
2 comprises a wetting agent.

1 26. The process of claim 25 wherein the wetting agent is selected
2 from the group consisting of nonionic fluorosurfactants, anionic fluorosurfactants,
3 ethoxylated tetramethyldecynediols, acetylenic glycol-based surfactants,
4 dialkylsulfosuccinates, and mixtures thereof.

1 27. The process of claim 25 wherein the wetting agent is a
2 fluoroaliphatic ethoxylates.

1 28. The process of claim 25 wherein the wetting agent is a present
2 in an amount of 0.1% to 1.0% of the weight of the dry film composition.

1 29. The process of claim 25 wherein the wetting agent is a present
2 in an amount of 0.1% to 0.5% of the weight of the dry film composition.

1 30. A liquid film lubricant comprising:
2 an oil; and
3 a surfactant,

4 wherein the liquid film lubricant has the characteristic that the
5 coefficient of friction is reduced when the liquid film lubricant is wetted as
6 compared to the coefficient of friction of the liquid film lubricant is unwetted.

1 31. The liquid film lubricant of claim 30, wherein the oil is selected
2 from the group consisting of vegetable oils, blown vegetable oils, polymers of
3 vegetable oils, animal oils, and blown animal oils. and mixtures thereof.

1 32. The liquid film lubricant 30, wherein the oil is selected from the
2 group consisting of blown canola oil, blown fish oil, canola oil, blown rapeseed oil,
3 naphthenic oil, and mixtures thereof.

1 33. The liquid film lubricant of claim 30, wherein the surfactant is
2 a non-ionic surfactant.

1 34. The liquid film lubricant of claim 33, wherein the surfactant is
2 selected from the group consisting of vegetable oil ethoxylates, ethoxylates of alkyl
3 alcohols, ethoxylates of acetylenic diols, block copolymers of ethylene and
4 propylene oxides, ethoxylates of alkyl carboxylates, alkyl polyglycosides and
5 mixtures thereof.

1 35. The liquid film lubricant of claim 33, wherein the surfactant is
2 present in an amount of about 0.1% to 10% of the total weight of the liquid film
3 lubricant.

1 36. The liquid film lubricant of claim 33, wherein the surfactant
2 is present in an amount of about 1.0% to 5% of the total weight of the liquid film
3 lubricant.

1 37. The liquid film lubricant composition of claim 33, wherein the
2 surfactant is present in an amount of about 2.5% of the total weight of the liquid film
3 lubricant.

1 38. A solid film lubricant comprising:
2 a wax; and
3 a surfactant,
4 wherein the solid film lubricant has the characteristic that the

5 coefficient of friction is reduced when the solid film lubricant is wetted as compared
6 to the coefficient of friction of the solid film lubricant is unwetted.

1 39. The solid film lubricant of claim 38, wherein the wax is
2 selected from the group consisting of carnauba wax, candelilla wax, montan wax,
3 microcrystalline waxes, solid alcohols, solid esters, and oxidized petroleum waxes.

1 40. The solid film lubricant of claim 38, wherein the wax is a
2 primary alcohols having at least 18 carbon atoms per molecule.

1 41. The solid film lubricant of claim 38, wherein the wax is an
2 ester of a primary alcohol having at least 18 carbon atoms per molecule with an
3 organic acid.

1 42. The solid film lubricant of claim 38, wherein the organic acid
2 is an unbranched monoacid, having at least 18 carbon atoms per molecule.

1 43. The solid film lubricant of claim 38, wherein the surfactant
2 is a non-ionic surfactant.

1 44. The solid film lubricant of claim 38, wherein the surfactant
2 is selected from the group consisting of vegetable oil ethoxylates, ethoxylates of
3 alkyl alcohols, ethoxylates of acetylenic diols, block copolymers of ethylene and
4 propylene oxides, ethoxylates of alkyl carboxylates, alkyl polyglycosides and
5 mixtures thereof.

1 45. The solid film lubricant of claim 38, wherein the surfactant
2 is present in an amount of about 0.05% to 10% of the total weight of the dry film
3 composition.

1 46. The solid film lubricant of claim 38, wherein the surfactant
2 is present in an amount of about 1.0% to 5% of the total weight of the solid film
3 lubricant.

1 42. The solid film lubricant of claim 38, wherein the surfactant
2 is present in an amount of about 1 % of the total weight of the solid film lubricant.

1 43. The solid film lubricant of claim 38 further comprising a
2 wetting agent.

1 44. The solid film lubricant of claim 43 wherein the wetting agent
2 is selected from the group consisting of nonionic fluorosurfactants, anionic
3 fluorosurfactants, ethoxylated tetramethyldecynediols, dialkylsulfosuccinates, and
4 mixtures thereof.

1 45. The solid film lubricant of claim 43 wherein the wetting agent
2 is a fluoroaliphatic ethoxylate.

1 46. The solid film lubricant of claim 43 wherein the wetting agent
2 is a present in an amount of 0.1% to 1.0% of the weight of the dry film
3 composition.

1 47. The solid film lubricant of claim 43 wherein the wetting agent
2 is a present in an amount of 0.1% to 0.5% of the weight of the dry film
3 composition.

1 48. A solid film lubricant comprising:
2 a wax; and
3 a wetting agent,
4 wherein the solid film lubricant has the characteristic that the

5 coefficient of friction is reduced when the solid film lubricant is wetted as compared
6 to the coefficient of friction of the solid film lubricant is unwetted.

1 49. The solid film lubricant of claim 48, wherein the wax is
2 selected from the group consisting of carnauba wax, candelilla wax, montan wax,
3 microcrystalline waxes, solid alcohols, solid esters, and oxidized petroleum waxes.

1 50. The solid film lubricant of claim 48, wherein the wax is a
2 primary alcohols having at least 18 carbon atoms per molecule.

1 51. The solid film lubricant of claim 48, wherein the wax is an
2 ester of a primary alcohol having at least 18 carbon atoms per molecule with an
3 organic acid.

1 52. The solid film lubricant of claim 48, wherein the organic acid
2 is an unbranched monoacid, having at least 18 carbon atoms per molecule.

1 53. The solid film lubricant of claim 48 wherein the wetting agent
2 is selected from the group consisting of nonionic fluorosurfactants, anionic
3 fluorosurfactants, ethoxylated tetramethyldecynediols, acetylenic glycol-based
4 surfactants, dialkylsulfosuccinates, and mixtures thereof.

1 54. The solid film lubricant of claim 48 wherein the wetting agent
2 is a fluoroaliphatic ethoxylate.

1 55. The solid film lubricant of claim 48 wherein the wetting agent
2 is present in an amount of 0.1% to 1.0% of the weight of the dry film
3 composition.

1 56. The solid film lubricant of claim 48 wherein the wetting agent
2 is present in an amount of 0.1% to 0.5% of the weight of the dry film
3 composition.